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(54) Title: SYNERGISTIC TERMITICIDAL COMPOSITION OF PYRETHROID AND N-PHENYL-PYRAZOLE			
(57) Abstract A termite control composition for soil treatment containing 3-cyano-1-(substituted phenyl) pyrazole derivative and a pyrethroid compound as effective ingredients, and a method of controlling pests such as termites using said composition. The termite control composition is improved for soil treatment as well as for the ability to prevent termites from passing through the pesticidally treated layer.			

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SYNERGISTIC TERMITICIDAL COMPOSITION OF PYRETHROID
AND N-PHENYL-PYRAZOTE

The present invention relates to a termite control composition for soil treatment containing a 3-cyano-1-(substituted phenyl)-pyrazole derivative and a
5 pyrethroid compound as effective ingredients.

The pyrazoles derivatives as effective ingredients of the termite control composition of the present invention are known compounds described in European patent application 295117 as well as in international patent
10 applications WO 93/6089 and 94/21606, which disclose that the compounds have a pesticidal effect on arthropods, vegetable nematodes, protozoan pests, and other pests. Many other pesticidal compounds can be used in combination with N-phenyl pyrazole derivatives. Pyrethroid compounds such as cyfluthrin, cypermethrin, deltamethrin, fenpropathrin, fenvalerate, and permethrin are
15 recited among many possibilities without any reference to any specific effect in any conditions.

A first object of the instant invention is to provide synergistic compositions of 3-cyano-1-(substituted phenyl)-pyrazole derivative.

Another object of the instant invention is to provide specific compositions
20 which have an improved activity against pests, especially against insects.

Another object of the instant invention is to provide specific compositions which have an improved activity against termites.

There are mainly two types of termite control methods : namely, wood application by applying a control agent to wood, and soil treatment by spraying
25 a control agent on the soil and/or under the floor. For existing houses, since the application of the agent to wood is rather difficult, soil treatment is generally used. In many cases, however, termites pass through the treated layer and eat the wood, and, therefore, it is desired to develop a termite control agent which has both the termite control effect and the ability to prevent termites from
30 passing through the pesticidally treated layer.

A further object of the instant invention is to provide a novel termite control composition (preferably for soil treatment) which has a strong termite control effect as well as the ability to prevent termites from passing through the pesticidally treated layer.

It has been found that these goals may be reached by mean of the compositions of the instant invention.

The compositions of the present invention comprise, as effective ingredients, a pyrethroid compound and a compound of formula (I)

5 1-[4-R¹ 2,6-(R²)_p phenyl] 3-cyano 4-[R⁴-S(O)_n] 5-R⁵ pyrazole (I)

wherein :

R¹ is halogen, lower haloalkyl, lower haloalkoxy or SF₅ (lower being an integer from 1 to 4, preferably one),

R² is halogen, the various R² being identical or different,

10 R⁴ is halogen, lower alkyl or haloalkyl,

R⁵ is halogen, lower alkyl or amino,

n is 0 or 1 or 2; p is 1 or 2 or 3 or 4, preferably 2.

Halo before the name of a radical means that this radical may be substituted by one or more halogen atoms.

15 A preferred compound of formula (I) is 5-amino-3-cyano-1-(2,6-dichloro-4-trifluoromethylphenyl)-4-trifluoromethylsulphonyl pyrazole.

The compositions of the invention comprise a synergistically amount of active ingredients.

20 Pyrethroid compounds which can be used in the present invention include all kind of pyrethroids, especially pyrethroids other than cyfluthrin, cypermethrin, deltamethrin, fenpropathrin, fenvalerate, and permethrin. Advantageously, pyrethroids which can be used in the invention are compounds selected from a group consisting of the following pyrethroid compounds, even tough not limited to these pyrethroid compounds:

- 25 1. Allethrin [dl-3-allyl-2-methyl-4-oxo-2-cyclopentenyl-dl- cis, trans-chrysanthemate]
2. Ethofenprox [2-(4-ethoxyphenyl)-2-methylpropyl-3-phenoxybenzyl ether]
3. Cycloprothrin [(RS)-a-cyano-3-phenoxybenzyl (RS)-2,2- dichloro-1-(4-ethoxyphenyl)-cyclopropane carboxylate]
- 30 4. Cyhalothrin [(RS)-a-cyano-3-phenoxybenzyl (Z)-(1RS, 3RS) 3-(2-chloro-3,3,3-trifluoro-1-propenyl)-2,2-dimethyl- cyclopropane carboxylate]
5. Cyfluthrin [(RS)-a-cyano-4-fluoro-3-phenoxybenzyl (1RS, 3RS)-(IRS, 3RS)-3-(2,2-dichlorovinyl)-2,2-dimethyl cyclopropane carboxylate]

6. Cypermethrin [(RS)-a-cyano-3-phenoxybenzyl (1RS, 3RS)-
(1RS,3SR)-3-(2,2-dichlorovinyl)-2,2-dimethyl cyclopropane carboxylate]
7. Pyrethrin
8. Tralomethrin [(S)-a-cyano-3-phenoxybenzyl (1R, 3S)-2,2-
5 dimethyl-3-(1,2,2,2-tetrabromoethyl)-cyclopropane carboxylate]
9. Fenvalerate [(RS)-a-cyano-3-phenoxybenzyl (RS)-2-(4-
chlorophenyl)-3-methylbutanoate]
10. Fenprothrin [(RS)-a-cyano-3-phenoxybenzyl-2,2,3,3-
tetramethyl cyclopropane carboxylate]
- 10 11. Flucythrinate [(RS)-a-cyano-3-phenoxybenzyl-(S)-2-(4-
difluoromethoxyphenyl)-3-methyl butylate]
12. Permethrin [3-phenoxybenzyl (1RS, 3RS)-(1RS, 3RS)-(2,2-
dichlorovinyl)-2,2-dimethyl cyclopropane carboxylate]
13. Bifenthrin [2-methylbiphenyl-3-yl-methyl (Z)-(1RS, 3RS)-3-(2-
15 chloro-3,3,3-trifluoroprop-1-enyl)-2,2-dimethyl cyclopropane carboxylate]
14. Silafluofen [4-ethoxyphenyl-[3-(3-phenoxy-4- fluorophenyl)
propyl](dimethyl) silane]
15. Lesmethrin [5-benzyl-3-furylmethyl dl-cis, trans- chrysanthemate]
16. Tefluthrin [2,3,5,6-tetrafluoro-4-methylbenzyl-(1RS)- cis-3-(Z-2-
20 chloro-3,3,3-trifluoroprop-1-enyl)-2,2-dimethyl cyclopropane carboxylate]
17. Acrinathrin [(S)-a-cyano-3-phenoxybenzyl (Z)-(1R, 3S)-2,2-
dimethyl-3-[2-(2,2,2-trifluoro-1-trifluoromethyl ethoxycarbonyl) vinyl]
cyclopropane carboxylate]
18. Prarethrin [(RS)-2-methyl-4-oxo-3-prop-2-enylcyclopent- 2-enyl
25 (1RS)-cis, trans-2,2-dimethyl-3-(2-methyl prop-1-enyl) cyclopropane
carboxylate]
19. Cismethrin [5-benzyl-3-furylmethyl (1R)-trans-2,2- dimethyl-3-(2-
methyl prop-1-enyl) cyclopropane carboxylate]
20. d-Phenothrin [3-phenoxybenzyl (1RS)-cis, trans-2,2- dimethyl-3-
30 (2-methyl prop-1-enyl) cyclopropane carboxylate]
21. Deltamethrin [(S)-a-cyano-3-phenoxybenzyl (1R)-cis-3- (2,2-
dibromovinyl)-2,2-dimethyl cyclopropane carboxylate]

22. Tetramethrin [cyclohex-1-ene-1,2-dicarboximide methyl (1RS, 3RS, 1RS, 3SR)-2,2-dimethyl-3-(2-methyl prop-1-enyl) cyclopropane carboxylate].

5 The synergistic compositions of the invention are compositions wherein the ratio by weight of the pyrethroid compound to the compound of formula (I) is between 0.1 and 10, preferably between 0.5 and 5.

The synergistic compositions of the invention are compositions, which are most useful for termite control, preferably with soil treatment. Thus, the
10 invention is also directed to a method of control of pests, especially of termites which comprises applying an effective amount of the compositions according to the invention, as herein described.

The liquid compositions of the invention generally comprise 0.001 to 50 % (all percentages are by weight in the instant specification, unless specifically
15 indicated otherwise) of compound of formula (I), preferably from 0.005 % to 10. The concentrated composition which are those used for storage and commercial purpose comprise generally from 1 to 20 % of this compound of formula (I).

When the compositions for soil treatment of the present invention are used
20 for termite control, especially for soil treatment and/or for treating under-floor soil, the quantity of the effective ingredient may be within a range between 0.01 g and 7 g, preferably between 0.1 g and 5 g per square meter. For wood treatment, such as timber or all kind of wood, the method of control of pest, especially of termites, according to the invention is impregnating the wood by
25 mean of a composition as herein before defined.

The application of the compositions of the invention to animals is generally made at 0.1 to 100 mg, preferably at 2 to 20 mg per kilogram of body weight of the animal.

The termite control composition of the present invention has a highly
30 significant termite control effect on houses damaging termites, for example Coptotermes formosanus, (Shiraki), Reticulitermes speratus (Kolbe), Odontotermes formosanus (Shiraki), and Cryptotermes domesticus (Haviland), as well as the ability to prevent termites from passing through pesticidally treated materials. The composition may be applied to or adsorbed in building

materials, furniture, leather, fabrics, vinyl coated articles, electric wires, or cables.

For the efficient use of the termite control composition for material or soil treatment of the present invention, the composition may be dissolved, suspended, mixed, adsorbed, or adhered on an appropriate solid and/or liquid vehicles (this word is used as a synonym of "carrier") according to the formulation generally used, together with auxiliary agents if required. This composition may be formulated into forms suited to the object of use, for example, an oil solution, emulsion, water solution, powder, granules, wettable powder, aerosol, smoking agent, or flowable agent.

Solid vehicles used in the present invention include, for example, clays such as kaolin, bentonite, and acid clay; talc materials such as talc and pyrophyllite; siliceous materials such as diatomaceous earth, silica sand, mica, synthetic silicates, and high dispersion synthetic silicates; and inorganic mineral powders such as pumice and sand. Liquid vehicles include, for example, alcohols such as methanol, ethanol, and ethylene glycol; ketones such as acetone, methylethyl ketone, and cyclohexanone; ethers such as ethyl ether, dioxane, tetrahydrofuran, and cellosolve; aliphatic hydrocarbons such as kerosene; aromatic hydrocarbons such as benzene, toluene, xylene, solvent naphtha, cyclohexane, and methyl naphthalene; and halogenated hydrocarbons such as chloroform, carbon tetrachloride, and chlorobenzene. These solid or liquid vehicles may be used alone or in combination.

Auxiliary agents used in the present invention include propellants, surface-active agents, fixing agents, dispersing agents, thickening agents, and bonding agents. Propellants include, for example, liquefied petroleum gas, dimethyl ether, and fluorocarbons. Surface-active agents include, for example, polyoxyethylene alkylaryl ether, polyoxyethylene sorbitane monolaurate, alkylallyl sorbitane monolaurate, alkylbenzene sulfonate, alkylnaphthalene sulfonate, lignin sulfonate, and sulfuric acid ester salts of higher alcohols. These surface-active agents may be used alone or in combination.

Fixing agents, dispersing agents, thickening agents, and bonding agents include, for example, casein, gelatine, starch, carboxymethyl cellulose, alginic acid, agar, polyvinyl alcohol, polyethylene glycol, polysodium acrylate, gum arabic, and xanthane gum, which may be used if required.

The termite control composition for soil treatment of the present invention may contain co-operating agents such as sinepyrin 500, piperonyl butoxide, and S-421.

5 The termite control composition of the present invention may be used not only for treating the surface or the interior of surrounding soil or under-floor soil for protecting wood such as trees, fences, and railroad ties, or buildings such as houses, warehouses, and industrial plants, but also in timber products such as plywood and furniture, wood products such as particle boards and half boards, and vinyl products such as coated wires and sheets.

10 The present invention also includes the aspects for preventive uses in places where the breeding of termites is expected as well as the above aspects.

Emulsifying agents which may be used are one or more of those selected from non-ionic or anionic emulsifying agents. Examples of non-ionic emulsifying agents which may be mentioned include
15 polyoxyethylenealkylphenylether, polyoxyethylenealkylether, polyethyleneglycol fatty ester, sorbitan fatty ester, polyoxyethylene sorbitan fatty ester, polyoxyethylenesorbitol fatty ester, polyoxyethylenepolyoxypropylenealkylether. Examples of anionic emulsifying agents which may be mentioned include alkyl sulphates, polyoxyethylenealkylether sulphates,
20 sulfosuccinates, taurine derivatives, sarcosine derivatives, phosphoric esters, alkylbenzenesulfonates and the like. A mixture consisting of polyoxyethylenestyrylphenylether and calcium alkylbenzenesulfonate is preferred. These emulsifying agents may be used in an amount of 5 to 20 weight parts per 100 weight parts of the composition of the present invention.

25 Compositions of the present invention may be prepared by any of conventional procedures suitable for emulsifiable concentrates.

The present invention is illustrated by the following examples, comparative examples and experimental examples, but is not limited to the details thereof.

30

EXAMPLES

Typical embodiments and test examples of the present invention will be shown below, but the present invention is not limited to these embodiments.

- In the description of these embodiments, the term "part(s)" means part(s) by weight. The test method of embodiments was in accordance with Japan Wood Preservation Association Standards No. 13, 1987, "Standards for Testing Methods of Termite Controlling Effects and Performance of Termite Controlling Agents for Soil Treatment (I)."

	Embodiment 1	
	Compound A	8.00 parts
	Bifenthrin	2.00 parts
10	Propylene glycol	5.00 parts
	Anionic surface-active agent	1.00 part
	Non-ionic surface-active agent	5.00 parts
	Xanthane gum	0.25 parts
	Silicone defoaming agent	0.50 parts
15	Water	78.25 parts

The above materials are uniformly mixed and suspended to form a flowable agent.

20	Embodiment 2	
	Compound A	1.00 part
	Bifenthrin	0.40 parts
	Propylene glycol	5.00 parts
	Anionic surface-active agent	1.00 part
25	Non-ionic surface-active agent	5.00 parts
	Xanthane gum	0.40 parts
	Silicone defoaming agent	0.50 parts
	Water	86.70 parts

- 30 The above materials are uniformly mixed and suspended to form a flowable agent.

	Embodiment 3	
	Compound A	4.00 parts

Permethrin	20.00 parts
Anionic surface-active agent	10.00 parts
N-methyl-2-pyrrolidone	10.00 parts
Aromatic solvent	56.00 parts

5

The above materials are uniformly dissolved to form an emulsion.

Test example 1

10 A testing apparatus was used in which two glass cylinders (each about 5 cm in diameter and about 12 cm in height) are connected at about 2 cm from the bottom with a glass tube about 1.5 cm in diameter and about 10 cm in length (graduated at 5-mm intervals for 5 cm at the center). The one glass cylinder was filled with about 60 g of non- treated soil adjusted to a moisture content of about 25%, and the other glass cylinder was filled with about 0.29 g of filter
15 paper (5.5 mm in diameter). The glass tube was filled, at a thickness of 1 cm, with test soil prepared by mixing 2.4 g of non- treated sandy soil which had passed through a 20-mesh screen and had been dried at 60°C until a constant weight had been achieved, with 0.45 g of the solution of the test composition of a predetermined concentration, and allowing the mixture to stand for 3 weeks in
20 a room without weather resistance treatment. The glass tube was connected to the glass cylinders.

In the glass cylinder filled with non treated soil, placed were 200 workers and 20 soldiers of *Coptotermes formosanus Shiraki*, and the testing apparatus was kept at a constant temperature chamber controlled at a temperature of 28°C,
25 and a relative humidity of 70% or higher.

The bored depth (millimetres = mm), damage by eating, and the termite control effect were determined 14 days after insects were put in place, and the effect was evaluated in accordance with the following criteria:

Damage by eating:

- 30 + 10% or less compared with non treatment
++ 11-50% or less compared with non treatment
+++ 51% or more compared with non treatment

Termite control effect:

- A 100% lethal

- B 80-99% lethal
- C 50-79% lethal
- D 49% lethal or less

5 Results are shown in Table 1 :

	Test composition	Concentration (%)	Bored depth (mm) 14 DAT	Damage by eating 14 DAT	Termite control effect 14 days later
Em-bodi-ments	compound A + bifenthrin	0.01 + 0.01	3	None	A
		0.01 + 0.005	2	None	A
		0.005 + 0.01	3	None	A
		0.005 + 0.005	7	None	A
		0.0025 + 0.01	5	None	A
		0.0025 + 0.005	9	None	A
		0.00125 + 0.01	7	None	A
		0.00125 + 0.005	9	None	A
Em-bodi-ments	Compound A + fenvalerate	0.02 + 0.05	2	None	A
		0.005 + 0.05	6	None	A
		0.02 + 0.01	1	None	A
		0.005 + 0.01	3	None	A
	Compound A + cypermethrin	0.02 + 0.025	0	None	A
		0.005 + 0.025	0	None	A
		0.02 + 0.005	2	None	A
		0.005 + 0.005	3	None	A
	Compound A + permethrin	0.02 + 0.1	0	None	A
		0.005 + 0.1	6	None	A
		0.02 + 0.02	8	None	A
	Compound A + tralomethrin	0.02 + 0.01	6	None	A
		0.005 + 0.01	4	None	A
		0.02 + 0.002	2	None	A
		0.005 + 0.002	8	None	A
	Compound A + fluvalinate	0.02 + 0.05	8	None	A
	Compound A + cyfluthrin	0.02 + 0.025	6	None	A
		0.005 + 0.025	6	None	A
		0.02 + 0.005	6	None	A

Compound A + ethofenprox	0.02	+	0.1	2	None	A
	0.005	+	0.1	8	None	A
	0.02	+	0.02	4	None	A
	0.005	+	0.02	7	None	A
Compound A + silafluofen	0.02	+	0.05	6	None	A
	0.005	+	0.05	5	None	A
	0.02	+	0.01	7	None	A

	Test composition	Concentration (%)	Bored depth (mm) 14 days later	Damage by eating 14 days later	Termite control effect 14 days later
Com- parati- ve exam- ples	Compound A	0.02	> 10	+	A
		0.01	> 10	+	A
		0.005	> 10	++	A
	Bifenthrin	0.01	> 10	+++	D
		0.005	> 10	+++	D
	Fenvalerate	0.05	> 10	+	A
		0.01	> 10	+++	D
	Cyperme-thrin	0.025	> 10	None	D
		0.005	> 10	+++	D
	Permethrin	0.1	> 10	None	D
		0.02	> 10	+++	D
	Tralomethrin	0.01	> 10	+++	D
		0.002	> 10	+++	D
	Fluvalinate	0.05	> 10	+++	D
	Cyfluthrin	0.025	> 10	None	D
		0.005	> 10	+++	D
	Ethofenprox	0.1	> 10	None	D
		0.02	> 10	+++	D
	Silafluofen	0.05	> 10	+	A
		0.01	> 10	+++	D
	Non-treatment		> 10	+++	D

Claims:

- 1) A pesticidal composition which comprises, as effective ingredients, a pyrethroid and a compound of formula (I)
5 1-[4-R¹ 2,6-(R²)_p phenyl] 3-cyano 4-[R⁴-S(O)_n] 5-R⁵ pyrazole (I)
wherein R¹ is halogen, lower haloalkyl, lower haloalkoxy or SF₅ (lower being an integer from 1 to 4, preferably one); R² is halogen, the various R² being identical or different; R⁴ is halogen, lower alkyl or haloalkyl; R⁵ is halogen, lower alkyl or amino; n is 0 or 1 or 2; p is 1 or 2 or 3 or 4, preferably
10 2.
- 2) A composition according to claim 1 wherein the effective ingredients are present in the composition in a synergistically amount.
- 3) A composition according to any one of claim 1 or 2 wherein the ratio by weight of pyrethroid compound to compound of formula (I) is between
15 0.1 and 10, preferably between 0.5 and 5.
- 4) A composition according to any one of claim 1 to 3 which comprises between 0.001 % to 50 % of compound of formula (I), preferably between 0.005 and 10 %.
- 5) A composition according to any one of claim 1 to 4 which
20 comprises between 1 and 20 % of compound of formula (I).
- 6) A composition according to any one of claim 1 to 5 when used against termites.
- 7) A composition according to any one of claim 1 to 6 wherein the compound of formula (I) is the 5-amino-3-cyano-1-(2,6-dichloro-4-trifluoromethylphenyl)-4-trifluoromethylsulphonyl pyrazole.
25
- 8) A composition according to any one of claim 1 to 7 wherein the pyrethroid compound is other than cyfluthrin, cypermethrin, deltamethrin, fenpropathrin, fenvalerate, and permethrin
- 9) A composition according to any one of claim 1 to 7 wherein the
30 pyrethroid compound is selected from a group consisting of Allethrin, Ethofenprox, Cycloprothrin, Cyhalothrin, Cyfluthrin, Cypermethrin, Pyrethrin, Tralomethrin, Fenvalerate, Fenpropathrin, Flucythrinate, Permethrin, Bifenthrin, Silafluofen, Lesmethrin, Tefluthrin, Acrinathrin, Prarethrin, Cismethrin, d-Phenothrin, Deltamethrin, Tetramethrin.

10) A method for controlling and preventing termites from passing through a pesticidally treated layer of soil or material whereby the said layer is treated by mean of an affective amount of a composition according to any one of claim 1 to 9

5 11) A method for controlling termites by soil treatment using 0.1 g/m² to 5 g/m² of a termite control composition according to any one of claim 1 to 9

10 12) A method for controlling pests of animals wherein the animal is trated by mean of a composition according to any one of claim 1 to 9, the rate of application being preferably comprised between 0.1 and 100 mg per kilo of body weight of the animal, preferably between 2 and 20 mg/kg.

INTERNATIONAL SEARCH REPORT

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PCT/EP 95/00601

A. CLASSIFICATION F SUBJECT MATTER
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According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

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Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	EP,A,0 435 609 (RHONE POULENC AGRIC LTD) 3 July 1991 see column 1, line 22-50 ---	1-12
A	EP,A,0 295 117 (MAY & BAKER LTD.) 14 December 1988 Cited in the application. See: p. 5, l. 4, 5 and p. 7, l. 64-p. 8, l. 5 ---	1-12
A	WO,A,93 06089 (ICI PLC) 1 April 1993 Cited in the application. see page 8, line 13-34 ---	1-12
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☒ Patent family members are listed in annex.

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Date of the actual completion of the international search

11 July 1995

Date of mailing of the international search report

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INTERNATIONAL SEARCH REPORT

Internat. Application No.

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